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environmental restoration project

A Department of Energy Environmental Cleanup Program

Environmental Restoration Project
Standard Operating Procedure

for:

Operation of Siemens D-500 X-ray Diffractometers

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Revision Log

Revision No.	*Effective Date*	*Prepared By*	*Description of Changes*	*Affected Pages*
R0	03/16/92	David Bish	New procedure	all
R1	04/11/01	David Vaniman	Revised to update records management and to fit new format for SOPs.	all
Review	12/16/2003	Mark Thacker	Deemed adequate.	All

Operation of Siemens D-500 X-ray Diffractometers

Table of Contents

Operation of Siemens D-500 X-ray Diffractometers

1.0 PURPOSE

This Standard Operating Procedure (SOP) states the responsibilities and describes the methods, procedures, and documentation used to obtain X-ray powder diffraction data from the Siemens D500 X-ray powder diffractometers at the Los Alamos National Laboratory (Laboratory) ER Project.

2.0 SCOPE

This SOP is a manadatory document and shall be implemented by all ER Project participants when collecting X-ray diffraction data for the ER Project using the LANL EES Siemens D-500 diffractometers.

3.0 TRAINING

3.1 All users of this SOP are trained by reading the procedure, and the training is documented in accordance with QP-2.2.

3.2 The **Geology Task Leader** will monitor the proper implementation of this procedure and ensure that relevant team members have completed all applicable training assignments in accordance with QP-2.2.

4.0 DEFINITIONS

4.1 *Machine Custodian* — The person who is in charge of an instrument and is responsible for the maintenance and safety of the instrument. This person's name shall be posted on the front of the diffractometers.

4.2 *Group Environment, Safety, and Health Plan* — A health and safety plan that is specific to the research Group activities and has been approved by the appropriate Division-level health and safety representative.

5.0 BACKGROUND AND PRECAUTIONS

Note: This SOP is to be used in conjunction with an approved Group environment, safety, and health plan.

5.1 Powder X-ray diffraction is a method by which investigators can identify the minerals present in a rock and obtain quantitative information on their abundance's and physical properties.

5.2 Malfunctions of the equipment are readily apparent. For the diffractometers, one either obtains counts or no counts.

5.3 The diffractometers produce ionizing radiation using high voltage sources. However, the diffractometers are interlocked such that if the panels are all in place, risk to the operator is almost non-existent.

5.4 Only two possibilities (rare) for data corruption exist: 1) loss of X-ray flux; 2) occurrence of noise spikes. The first problem can be identified by the lack of any diffracted intensity, i.e., counts = 0. Such data should be discarded. The occurrence of the second possibility can be noted by "peaks" only one 2T step wide. Such data are generally usable and only need to be discarded when a noise spike overlaps on an important peak.

5.5 The person requesting XRD analyses will record of sample submittals and analysis results in their research notebook.

6.0 RESPONSIBLE PERSONNEL

The following personnel are responsible for activities identified in this procedure.

6.1 Focus Area Leader

6.2 Team Leader

6.3 Quality Program Project Leader

6.4 Geology Task Leader

6.5 ER Project personnel

7.0 EQUIPMENT

7.1 Siemens D500 X-Ray Powder Diffractometers

7.2 Materials Data Inc. Jade X-Ray Data Package

7.3 Materials Data Inc. DataScan Instrument Control Package

8.0 PROCEDURE

Note: ER Project personnel may produce paper copies of this procedure printed from the controlled-document electronic file located at http://erinternal.lanl.gov/home_links/Library_proc.htm. However, it is their responsibility to ensure that they are trained to and utilizing the current version of this procedure. The author may be contacted if text is unclear.

Note: Deviations from SOPs are made in accordance with QP-4.2, Standard Operating Procedure Development and documented in accordance with QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

Note: The machine custodian is responsible for both alignment and calibration of the diffractometers and the training of any potential users of the diffractometers. The geology task leader (TL) has the responsibility to assure correct implementation of this procedure for ER work. The TL may delegate performance of the procedure to any properly trained and certified individual.

8.1 Calibration

For Alignment and calibration, see SOP-9.04, Calibration and Alignment of the Siemens Diffractometers.

8.2 Control of Samples

Samples will be tracked, stored, shipped, and handled in accordance with SOP-9.01. Care shall be exercised to label all X-ray runs with correct sample name, checking sample output against names on the sample bottles.

8.3 Diffractometer Operation

6.3.1 Turn on diffractometer as outlined in the Siemens D500/501 Operating Instructions.

6.3.2 Insert sample and turn on X-rays either by pressing the shutter-open button or by placing shutter in automatic mode.

6.3.3 The instrument is operated and data analyses conducted using the MDI software packages.

8.4 System Shut-Down

The diffractometer is shut down in accordance with the instruction manual or the instructions listed on the front of the diffractometer.

8.5 Data Analysis

6.5.1 The data are regressed and displayed using the MDI software package.

6.5.2 Crystalline phases are identified by comparing their patterns with patterns of pure standards, patterns from the ICDD files, or with calculated patterns.

6.5.3 Quantitative X-ray diffraction analysis is conducted in accordance with the methods of FULLPAT (ref. Chipera and Bish, 2001).

8.6 Procedural Deviations

Deviations from this procedure and the effects it may have on the resulting work shall be documented as described in QP-4.2.

8.7 Documentation

6.7.1 All raw X-ray data stored on magnetic or optical media shall periodically be backed up onto magnetic tape and stored in a fireproof safe.

6.7.2 Records that are readily regenerated from the raw data such as hard copy plots and peak search data sheets may be placed in labeled three-ring binders.

8.8 Lessons Learned

During the performance of work, ER Project personnel shall identify, document and submit lessons learned in accordance with QP-3.2, Lessons Learned. This QP can be located at: http://erinternal.lanl.gov/home_links/Library_proc.htm.

9.0 REFERENCES

ER Project personnel may locate the ER Project Quality Management Plan/ER Project QP requirements crosswalk at http://erinternal.lanl.gov/home_links/Library_proc.htm.

The following documents have been cited within this procedure.

Chipera S. J. and D. L. Bish, "FULLPAT: A Full Pattern Quantitative Analysis Program for X-ray Powder Diffraction." User Manual, LANL XRD laboratory, 2001.

MDI (Materials Data Incorporated), DataScan 3.2, An Automated Control and Data Acquisition System for X-ray Diffractometers, 1995-1999.

MDI (Materials Data Incorporated), Jade 5, XRD Pattern Processing, 1991-1999.

Siemens D500/501 Operating Instructions, C72000-B3463-A42, Siemens Corporation, Cherry Hill, New Jersey.

QP-2.2, Personnel Orientation and Training

QP-3.2, Lessons Learned

QP-4.3, Records Management

QP-5.7, Notebook Documentation for Environmental Restoration Techncial Activities.

SOP-9.10, Sample Control and Field Documentation

SOP-9.04, Calibration and Alignment of the Siemens Diffractometers

10.0 RECORDS

The Geology Task Leader (TL) is responsible for submitting the following records (processed in accordance with QP-4.3) to the Records Processing Facility.

Notebook records of the sample handling and results of analysis relevant to production of X-ray diffraction data.

Data submittals for the ER electronic database.

Using a token card, click here to record "self-study" training to this procedure.

If you do not possess a token card or encounter problems, contact the RRES-ECR training specialist.